capacity. As the liquor level drops in the second effect, more liquor is fed from the first effect and fresh lye is fed to the first effect so as to keep the liquid levels in each effect constant. It is important for the smooth and uniform operation of the plant that feed to each effect be regular and steady rather than than that the level be allowed to drop and then a large charge fed in. With a little practice and care, the feed valves can be set so as to feed at the required uniform rate. Liquid level regulators can be used, but the salt content of spent soap lye is so high that these devices are not entirely reliable on soap lye evaporators. Liquid level controllers for the first effect are fairly satisfactory, but when used on the second effect they require so much attention that manual control is usually used.

Steam to the first effect is regulated as required to show a positive pressure on the condensate drain.

Water to the barometric condenser is adjusted to maintain a uniform vacuum in the second effect and uniform temperature in the tail pipe. The greatest amount of water will be required at the start of the run. As the concentration of the liquor increases and the rate of evaporation decreases, less condensing water will be required.

Separation of Salt

In concentrating spent soap lye to crude glycerine about 11 to 15 lbs. of salt is separated from each 100 lbs. of lye, the exact amount depending upon the amounts of salt and glycerine in the spent lye and on the concentration of the finished crude. As soon as the liquor in either effect reaches the salting point, salt will begin to crystallize and drop to the bottom of the evaporator. The presence of salt may be observed in the liquor which is thrown against the sight glasses on the vapor belt. After salt begins to crystallize out, the lye has no further value in dissolving any salt remaining on the tubes from a previous run, as it is then a supersaturated salt solution, and it is time to start removing the salt from the evaporators.

Methods of Recovering Salt

There are three generally used methods of separating the salt from the glycerine.

(This paper will be concluded in the November issue of OIL & SOAP.)

The Refractometric Determination of Iodine Number in Flaxseed Oils

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INTRODUCTION

As linseed oil is used primarily in the manufacture of paints, varnishes, and linoleum, the rapidity with which a given sample of oil will oxidize is usually the most important single factor governing its quality. Although it is true that the drying time of low-quality linseed oils may be materially shortened by the addition of chemical "driers," oils that have low initial drying times nevertheless produce paint films of better quality.

Iodine number as a measure of the total degree of unsaturation of the oil may be considered a quantitative measure of the quantity of oxygen the oil is potentially able to absorb. The rate of oxidation, however, depends not only upon the total degree of unsaturation but upon the relative proportions of the various unsaturated fatty acids in the glycerides of the oil. As the iodine number does not differentiate between these various unsaturated fatty acids, it may not be considered as an exact measure of the drying time of the oil. Rapid-drying raw linseed oils, however, are high in linolenic-acid content and show a high iodine number. Conversely, slow-drying linseed oils are relatively low in linolenic-acid content and have correspondingly low iodine numbers. The iodine-number determination may therefore be considered a good practical (though not exact) method for determining the relative drying times of untreated raw linseed oils, and as such is used extensively in commercial practice. The iodine numbers of linseed oils from different lots of commercial flaxseed range from about 150 to 200 or over. Oils having iodine numbers below 165 are generally considered to be of decidedly inferior quality whereas iodine numbers of 185 or over usually indicate superior drying properties.

The importance of iodine number in the linseed-oil manufacturing industry has greatly increased during recent years, because of the fact that large quantities of flaxseed have been marketed, the oil of which has possessed very inferior drying qualities and correspondingly low iodine numbers. The production of these large quantities of low-quality flaxseed has been due partly to drought conditions and unseasonably high temperatures in the principal flax-growing regions of the United States over a period of years; and partly to the introduction of new varieties of flax that have been bred for high yields and disease-resistant qualities, but which inherently produce low-quality oils.

Since such a wide variation exists in the iodine number of oils prepared from different lots of flaxseed, the linseed-oil industry should be benefited by a simple method for determining in advance the iodine number of the oil that could be pressed from a given lot of flaxseed.

Relationship Between Iodine Number and Refractive Index

It has long been known that in the case of animal and vegetable oils in general, a positive correlation exists between refractive index and iodine number. Lewkowitsch (4), however, after accumulating data on a large number of different oils concluded that no definite relationship existed between these two factors. Niegemann and Kayser (5), on the other hand, reported such a relationship in the case of oils from flaxseed samples grown in a given region. Arnold (1), and Backer (2), have demonstrated a relationship between iodine number, saponification number and refractive index.

Pickering Cowlishaw (6) have developed the following equation to show the relationship between refractive index, iodine number, saponification number, and acid number:

$$n_D^{40} = 1.4643 - 0.000066\ S - \frac{0.0096A}{S} + 0.000117\ I$$

where S = Saponification number
A = Acid number
I = Iodine number

This equation was shown to apply to the freshly prepared oils from various kinds of oil-bearing seeds. The equation does not hold, however, for oils that have been prepared for an appreciable length of time, for the oxidation and polymerization that take place on standing cause a marked increase in the refractive index.

In the case of flaxseed, the saponification number of the oil is relatively constant for all varieties and types of seed, and the acid value of the freshly pressed oil is uniformly low except in the case of oil from badly damaged seed. It should therefore be possible to determine the iodine number directly from the refractive index of the freshly prepared oil provided the oil is prepared in such a manner that no appreciable degree of oxidation or polymerization can take place.

Hopper[1] has determined the refractive indices and iodine numbers of expressed oils from 1,500 samples of flaxseed and has noted a very significant relationship between the two values. Geddes and Lehberg (3) obtained a correlation of +.647 between the refractive index and iodine number of oils extracted by diethyl ether, with a standard error of prediction for iodine number of ± 2.75.

EXPERIMENTAL PROCEDURE

Collection of Flaxseed Samples

For the purpose of the present investigation, 96 samples of flaxseed were obtained representing a great diversity of types and exhibiting a corresponding great diversity in physical and chemical characteristics. This assortment was represented by samples of the following types:

1. Domestic commercial flaxseed.
2. Canadian commercial flaxseed.
3. Indian commercial flaxseed.
4. Argentine commercial flaxseed.
5. Thirteen individual flaxseed varieties.
6. Samples grown experimentally in North Dakota, South Dakota, Minnesota, Kansas, California, Oregon, Wyoming, Arizona, Missouri, New Jersey, and Saskatchewan.
7. Immature flaxseed.
8. Frost-damaged flaxseed.
9. Scabby flaxseed.

The samples in this series showed the following ranges in physical and chemical characteristics:

1. Moisture content: 4 per cent to 16 per cent.
2. Oil content: 32.57 per cent to 45.66 per cent (dry basis).
3. Iodine number of oil (Wijs): 155.4 to 197.3.
4. Refractive index of oil at 25° C.: 1.47589 to 1.48065.

Preparation of Oil Samples

For the purpose of this study it was desired to obtain samples of oil from the flaxseed samples in an essentially unchanged condition. Cold pressed oils should most closely approximate this condition. But when large numbers of samples are to be handled a more convenient method of preparing the oil samples is desirable. A rapid partial extraction method was developed therefore which yielded oils essentially identical with the cold pressed oils.

In this method approximately 2 grams of the freshly ground sample is mixed with 20 ml. of petroleum ether,[2] and filtered into a small shallow evaporating dish. The bulk of the solvent is evaporated off on the steam bath and the oil is then placed in an air oven at 105° C. for 30 minutes.

A comparison of the refractive indices of oils from six varieties of flaxseed prepared by cold pressing in a laboratory hydraulic press and by the rapid partial extraction method is shown in table 1.

Most of the oils used in the present study were prepared by the rapid partial-extraction method. A few were prepared by pressing at room temperature at a pressure of approximately 16,000 pounds to the square inch.

Table 1.—Refractive indices at 25° C. of oils prepared by cold pressing and by the rapid partial-extraction method.

Sample number	Cold pressed oil n_D^{25}	Oil by rapid partial extraction n_D^{25}	Flaxseed variety
31....	1.47971	1.47970	New Golden
32....	1.47889	1.47894	N.D.R. 114
33....	1.47816	1.47814	Buda
34....	1.47657	1.47654	Walsh
35....	1.47617	1.47621	Bison
36....	1.47907	1.47905	Punjab

Determination of Iodine Number

Iodine numbers were determined by the Wijs method according to the instructions of the Federal Specifications Board for determining the iodine number of raw linseed oil. These instructions specify the use of 0.09 to 0.15g. samples, and a reaction time of one hour at a temperature of from 21° to 23° C. Duplicate determinations in most cases agreed within 0.3 unit in terms of iodine number.

Determination of Refractive Index

The refractive indices of the oils were determined with a dipping type refractometer equipped with interchangeable water-jacketed double-prism heads. With such an instrument (Fig. 1) the refractive index can be determined to an accuracy of ± 0.00002, using two or three drops of oil. Water at room temperature was passed through the water jackets, the temperature being read to the nearest 0.1° C. Refractive index readings in all cases were calculated to 25.0° C. by use of the formula:

$$n_D^{25} = n_D^T + K\ (T - 25)$$

where K = 0.000357
and T = temperature in °C. at

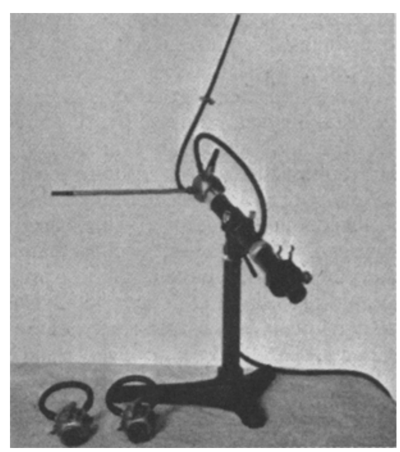

Fig. 1. Zeiss Dipping-Type Refractometer with Interchangeable Double-Prism Heads.

[1]Personal communication with Dr. T. H. Hopper, No. Dak. Agri. Expt. Sta., Fargo, N. D.

[2]For this purpose a petroleum ether conforming to the specifications adopted by the American Oil Chemists Society (7) should be used.

Table 2.—Iodine number (Wijs) and refractive index of oils from 96 samples of flaxseed.

Sample number	Description of sample	A Iodine number	n_D^{25}	B Iodine number from refractive index	Difference B - A
81	Bison, 1935; Moran, Kansas	155.4	1.47589	156.6	+1.2
74	Rio, 1934; Fargo, N. Dak	157.0	1.47582	156.1	—0.9
78	Rio, 1935; Moran, Kans	158.1	1.47627	159.9	+1.8
35	Bison	159.3	1.47617	159.0	—0.3
5	Bison; Missouri	161.5	1.47653	162.1	+0.6
106	Bison, 1935; Sheridan, Wyo	161.9	1.47657	162.5	+0.6
7	Bison, 1934	162.1	1.47644	161.4	—0.7
34	Walsh	162.2	1.47657	162.5	+0.3
70	Bison, 1934; Fargo, N. Dak	162.5	1.47651	162.0	—0.5
84	Bison, 1935; Dickinson, N. Dak	164.4	1.47686	165.0	+0.6
1	Bison; Arthur, N. Dak	165.5	1.47679	164.4	—1.1
79	Linota, 1935; Moran, Kans	166.0	1.47708	166.8	+0.8
85	Bison, 1935; Morris, Minn	166.4	1.47708	166.8	+0.4
38	Rio	167.9	1.47713	167.3	—0.6
105	Rio, 1935; Sheridan, Wyo	168.0	1.47713	167.3	—0.7
73	Buda, 1934	170.2	1.47747	170.2	0.0
83	Bison, 1935; New Brunswick, N. J.—Scabby	171.0	1.47743	169.9	—1.1
89	Domestic Commercial, 1934	171.2	1.47756	171.0	—0.2
93	Domestic Commercial, 1934	172.5	1.47779	172.9	+0.4
107	Redwing, 1935; Sheridan, Wyo	172.6	1.47775	172.6	0.0
80	Redwing, 1935; Moran, Kans	172.6	1.47790	173.9	+1.3
111	Bison, 1935; Newell, S. Dak	172.9	1.47773	172.4	—0.5
108	Linota, 1935; Sheridan, Wyo	173.7	1.47789	173.8	+0.1
6	Domestic Commercial	173.9	1.47790	173.9	0.0
71	Linota, 1934; Fargo, N. Dak	174.1	1.47809	175.5	+1.4
2	Bison	174.4	1.47780	173.0	—1.4
110	Rio, 1935; Newell, S. Dak	174.5	1.47796	174.4	—0.1
10	Domestic Commercial	174.6	1.47789	173.8	—0.8
4	Punjab; California	175.2	1.47809	175.5	+0.3
8	Domestic Commercial	175.5	1.47814	176.0	+0.5
33	Buda	176.1	1.47815	176.1	0.0
46	Punjab, 1935; Davis, Calif	176.1	1.47819	176.4	+0.3
82	Linota, 1935; New Brunswick, N. J.—Scabby	176.2	1.47814	176.0	—0.2
88	Domestic Commercial, 1934	177.8	1.47819	176.4	—1.4
87	Domestic Commercial, 1934	179.1	1.47852	179.2	+0.1
65	Indian, 1934; Brawley, Calif	179.2	1.47863	180.2	+1.0
72	Redwing, 1934; Fargo, N. Dak	179.5	1.47853	179.3	—0.2
64	Indian, 1934; Calexico, Calif.	179.6	1.47845	178.6	—1.0
41	Punjab, 1935; Davis, Calif	179.8	1.47859	179.8	0.0
112	Redwing, 1935; Newell, S. Dak	180.0	1.47860	179.9	—0.1
42	Punjab, 1935; Davis, Calif	180.1	1.47851	179.1	—1.0
48	Punjab, 1935; Shafter, Calif	180.2	1.47869	180.7	+0.5
43	Punjab, 1935; Willows, Calif	180.4	1.47869	180.7	+0.3
59	Indian, 1935; Brawley, Calif	180.7	1.47871	180.9	+0.2
54	Punjab, 1935; Imperial, Calif	180.9	1.47877	181.4	+0.5
40	Linota	181.0	1.47869	180.7	—0.3
44	Punjab, 1935; Concord, Calif	181.0	1.47866	180.4	—0.6
92	Domestic Commercial, 1934	181.3	1.47889	182.4	+1.1
39	Redwing	181.3	1.47872	181.0	—0.3
90	Domestic Commercial, 1934	181.4	1.47882	181.8	+0.4
60	Indian, 1934; Holtville, Calif.	181.5	1.47889	182.4	+0.9
32	N. D. R. 114	181.7	1.47889	182.4	+0.7
37	Bolley's Golden	181.8	1.47865	180.4	—1.4
11	Imported Indian, commercial	182.0	1.47897	183.1	+1.1
69	No. 4 Canadian Western	182.0	1.47900	183.3	+1.3
47	Abyssinian, 1935; Davis, Calif	182.1	1.47891	182.6	+0.5
57	Punjab, 1935; El Centro, Calif	182.4	1.47893	182.7	+0.3
113	Linota, 1935; Newell, S. Dak	182.6	1.47874	181.1	—1.5
68	No. 2 Canadian Western	182.8	1.47898	183.1	+0.3
53	Punjab, 1935; Holtville, Calif	183.0	1.47905	183.8	+0.8
101	Rio, 1935; Union, Ore	183.8	1.47918	184.9	+1.1
12	Imported Argentine, commercial	183.9	1.47903	183.6	—0.3
76	Imported Argentine, commercial	184.0	1.47899	183.2	—0.8
36	Punjab	184.0	1.47907	183.9	—0.1
67	No. 1 Canadian Western, 1934	184.2	1.47902	183.5	—0.7
61	Punjab, 1935; Holtville, Calif	184.7	1.47918	184.9	+0.2
56	Punjab, 1935; Holtville, Calif	185.2	1.47912	184.4	—0.8
86	Domestic Commercial, 1934	185.2	1.47916	184.7	—0.5
77	Punjab, 1935; Yuma, Ariz	185.5	1.47926	185.6	+0.1
55	Punjab, 1935; Brawley, Calif	185.8	1.47923	185.3	—0.5
58	Punjab, 1935; Calipatria, Calif	186.3	1.47933	186.2	—0.1
52	Punjab, 1935; Calexico, Calif	186.3	1.47940	186.8	+0.5
115	Bison, 1935; Saskatoon, Sask.	188.3	1.47960	188.5	+0.2
49	Punjab, 1935; Madera, Calif	188.5	1.47946	187.3	—1.2
66	No. 1 Canadian Western	188.5	1.47955	188.1	—0.4
31	New Golden	188.6	1.47971	189.4	+0.8
96	Unknown	188.6	1.47973	189.6	+1.0
114	Rio, 1935; Saskatoon, Sask	188.9	1.47949	187.5	—1.4
45	Punjab, 1935; Rio Vista, Calif	188.9	1.47957	188.2	—0.7
98	Unknown	190.5	1.47971	189.4	—1.1
3	Abyssinian; California	190.5	1.47982	190.4	—0.1
103	Redwing, 1935; Union, Ore	190.7	1.47987	190.8	+0.1
102	Bison, 1935; Union, Ore	190.9	1.47992	191.3	+0.4
100	Unknown	190.9	1.47992	191.3	+0.4
51	Heavy frost damage and scabby	191.0	1.47981	190.3	—0.7
97	Unknown	191.8	1.48010	192.8	+1.0
99	Unknown	191.9	1.48004	192.3	+0.4
104	Linota, 1935; Union, Ore	192.7	1.48010	192.8	+0.1
62	Abyssinian, 1935; Meloland, Calif.	193.2	1.48016	193.3	+0.1
95	Unknown	193.4	1.48024	194.0	+0.6
50	Immature	194.6	1.48017	193.4	—1.2
116	Redwing, 1935; Saskatoon, Sask	194.6	1.48030	194.5	—0.1
117	Linota, 1935; Saskatoon, Sask	194.9	1.48047	196.0	+1.1
91	Domestic Commercial, 1934	195.2	1.48021	193.7	—1.5
109	Redwing, 1935; Edmonton, Alberta—Immature	196.0	1.48053	196.5	+0.5
63	Abyssinian, 1935; Heber, Calif	197.3	1.48065	197.5	+0.2

which reading was taken.

Discussion of Results

The correlation coefficient between the iodine numbers and refractive indices of the series of samples under investigation (table 2) was calculated to be +.9965 with a standard error of prediction for iodine number of ± 0.82. The linear relationship between the two values is expressed by the regression equation:

$$I = 8584.97\ n_D^{25} - 12513.83.$$

where I = Wijs iodine number.

The regression line is shown graphically in Fig. 2, page 256.

The iodine numbers on the same series of samples calculated from this equation are listed in column B of table 2. Comparing these values with the values determined directly by the Wijs method (Column A), the maximum and average errors for the refractometric method are seen to be 1.8 and 0.6 units, respectively, in terms of iodine number. The maximum and average percentage errors are 1.1 per cent and 0.3 per cent, respectively. Errors of this magnitude are well within the range of the experimental error usually encountered in the determination of iodine number.

Limitations of the Method

From the above regression equation a table has been prepared (table 3) for converting refractive index readings to Wijs iodine numbers. It should be distinctly understood that this method for determining iodine number is meant to apply only to samples of oil prepared from flaxseed in such a way that no significant amount of hydrolysis, oxidation, or polymerization occur, and no solvent residues remain in the oil. Such samples may be prepared either by cold pressing or by the rapid partial-extraction method herein described. Immaturity, frost damage, or scabbiness of the seed do not appear to affect the reliability of the method. The method is not reliable, however, for oils from flaxseed that have become fermented or musty because of prolonged storage at high moisture content. Such flaxseed may readily be recognized by its sour or musty odor. Oils from such seed will generally be found to have undergone considerable hydrolysis.

The method may not be used for the direct determination of iodine number of commercial linseed oils since the processing that the oil undergoes tends to alter its refractive index.

Value of the Method

The principal value of the method should be for determining in advance the iodine number of the linseed oil which a given lot of flaxseed will produce. The method should be of considerable value to the flaxseed crusher because determinations may be made in a small fraction of the time required

Table 3.—Conversion table for determining Wijs iodine number of freshly prepared flaxseed oil from refractive index. Data calculated from regression equation:

$$I = -12513.827 + 8584.966 n_D^{25}$$

n_D^{25}	Iodine number	n_D^{25}	Iodine number	n_D^{25}	Iodine number	n_D^{25}	Iodine number
1.4750	149.0	1.4767	163.6	1.4784	178.2	1.4801	192.8
1.4751	149.9	1.4768	164.5	1.4785	179.0	1.4802	193.6
1.4752	150.7	1.4769	165.3	1.4786	179.9	1.4803	194.5
1.4753	151.6	1.4770	166.2	1.4787	180.8	1.4804	195.4
1.4754	152.4	1.4771	167.0	1.4788	181.6	1.4805	196.2
1.4755	153.3	1.4772	167.9	1.4789	182.5	1.4806	197.1
1.4756	154.1	1.4773	168.7	1.4790	183.3	1.4807	197.9
1.4757	155.0	1.4774	169.6	1.4791	184.2	1.4808	198.8
1.4758	155.9	1.4775	170.5	1.4792	185.1	1.4809	199.6
1.4759	156.7	1.4776	171.3	1.4793	185.9	1.4810	200.5
1.4760	157.6	1.4777	172.2	1.4794	186.8	1.4811	201.4
1.4761	158.4	1.4778	173.0	1.4795	187.6	1.4812	202.2
1.4762	159.3	1.4779	173.9	1.4796	188.5	1.4813	203.1
1.4763	160.2	1.4780	174.8	1.4797	189.3	1.4814	203.9
1.4764	161.0	1.4781	175.6	1.4798	190.2	1.4815	204.8
1.4765	161.9	1.4782	176.5	1.4799	191.1	1.4816	205.7
1.4766	162.7	1.4783	177.3	1.4800	191.9	1.4817	206.5

for the conventional iodine number determinations, and because the use of high-priced chemical reagents is eliminated.

The plant breeder should also find the method helpful when requiring the iodine numbers of the oils from very small samples of seed. The refractometric determination of iodine number requires only about 0.03 g. of oil, which may be obtained from about 0.08 g. of flaxseed.

SUMMARY

In a study of 96 diversified samples of flaxseed a correlation coefficient of +.9965 between the Wijs iodine number and refractive index of the freshly prepared oils was found, the standard error of prediction for iodine number being ± 0.82.

From the regression equation obtained from the same data a table has been prepared for converting refractive index readings into Wijs iodine numbers. The method is applicable only to freshly prepared cold pressed oils or their equivalent from reasonably sound flaxseed.

The refractometric method for determining the iodine number of flaxseed oils should be useful to the flaxseed crusher and to the plant breeder.

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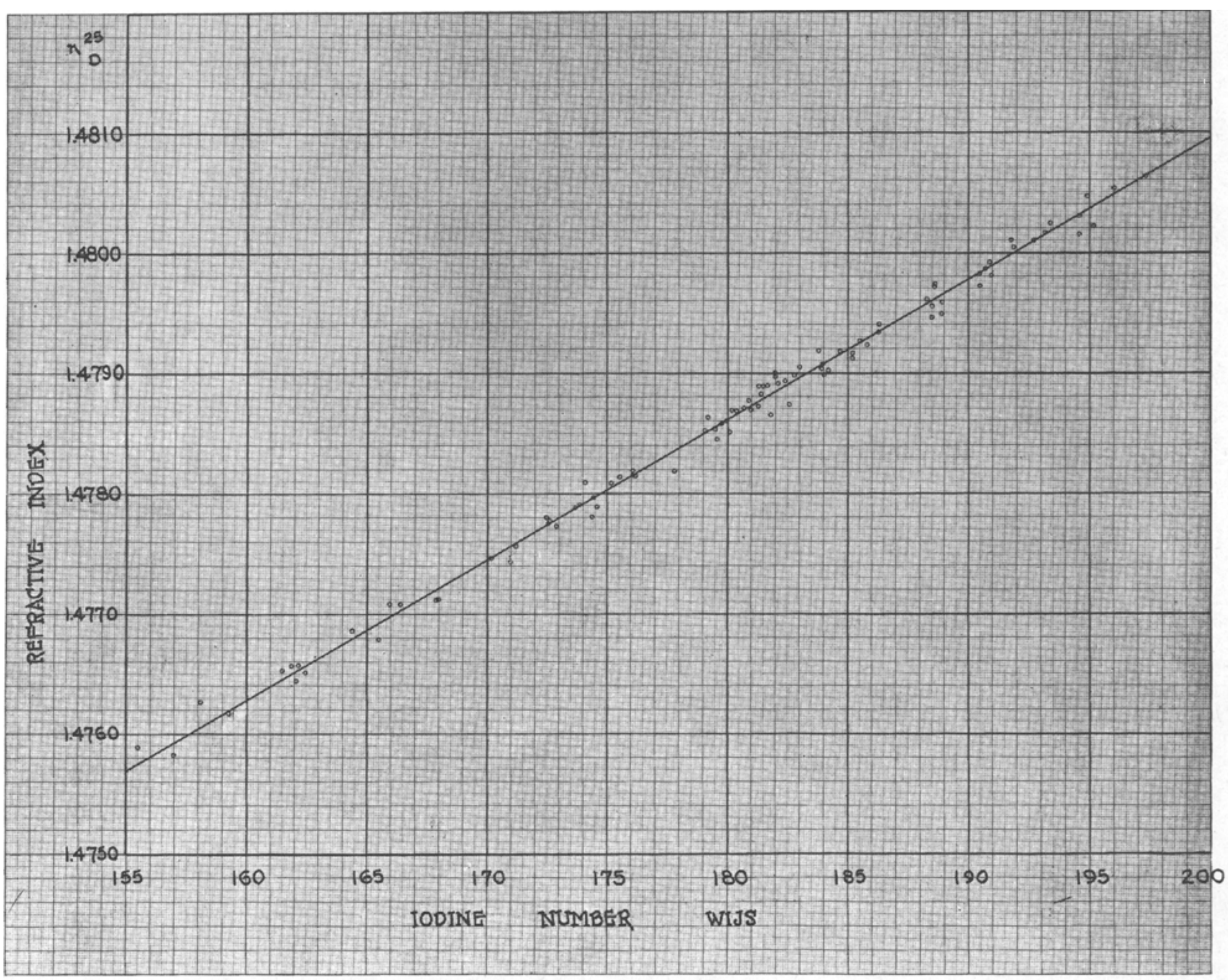

Fig. 2. Refractive Indices and Iodine Numbers of Oils from 96 Samples of Flaxseed.